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भारतीय मानक

सिन्टैन — परीक्षण पद्धति

Indian Standard

SYNTANS — METHODS OF TEST

UDC 675·024·47 : 620·1

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Tanning Materials and Allied Products Sectional Committee, CHD 018

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Tanning Materials and Allied Products Sectional Committee had been approved by the Chemical Division Council.

Syntans are water soluble water dispersible high molecular mass synthetic organic compounds meant for different multiple end-uses in tanning/leather industry. These syntans are used for pretanning, retanning, bleaching, mellowing, filling, mordanting for dyeing, neutralising, to tighten grain, minimising sludge in vegetable tannin liquors, etc. These syntans are not structurally related to natural vegetable tannins and sometimes are complexed with metals like chromium, aluminium and other metals. As a result of this structural dissimilarity, the word 'SYNTAN' is used to define these synthetic tanning agents.

Syntans help to improve the various physical, chemical and antimicrobial properties of leathers. The type of syntan used has a considerable effect on the resultant leather. Some of the syntans are introduced mainly to substitute or supplement natural vegetable tannins. In the case of syntans meant for purposes other than tanning like syntans for bleaching. filling, etc, the criteria of converting animal hide or skin into leather, and thereby increasing shrinkage temperature of leather, are not required. Also syntans meant for filling should not have film forming properties in the leather, for example, binders.

In reporting the results of a test or analysis, made in accordance with this standard if the final value, observed or calculated is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

SYNTANS — METHODS OF TEST

1 SCOPE

This standard prescribes methods of test for syntans of all kinds in the form of solid and liquid.

2 REFERENCE

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
265 : 1987	Hydrochloric acid (*third revision*)
1070 : 1977	Water for general laboratory use (*second revision*)
1040 : 1960	Glossary of terms relating to hides, skins and leather
1839 : 1961	Toluene, reagent grade

3 TERMINOLOGY

3.1 For the purpose of this standard, definitions given in IS 1640 : 1960 and the following shall apply.

3.1.1 *Total Solids*

The amount of solid material contained in any syntan.

3.1.2 *Total Solubles*

The total amount of water soluble material present in any syntan.

3.1.3 *Insolubles*

The amount of material present in any syntan that is not soluble in water. This is obtained from the difference between the percentage of total solid and total solubles.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

5 PREPARATION OF SAMPLE FOR TEST

5.1 Spray Dried Power — Mix thoroughly and sample.

5.2 Liquid Syntan

Mix thoroughly so as to include any sediment which may be present in the liquid sample.

6 PREPARATION OF SOLUTION

6.1 Quantity of Material to Employ

Weigh out such a quantity of the material which will give as nearly as possible 4·0 g per litre of tannin matter absorbable by hide powder, but in any case not less than 3·75 g, and not more than 4·25 g

NOTE — In the event of the results of an analysis showing a tanning strength outside these limits, the analysis shall be repeated employing the required amount of material.

6.2 Solid Syntan

Weigh out such a quantity of the material which will give as nearly as possible 1 litre of solution of required analytical strength (*see* **6.1**). Wash into a beaker with boiling water, stir, and transfer the dissolved portion to a 1 000-ml flask. Repeat the process until all the material is dissolved and the flask contains about 500 ml of solution. Cool and make up the volume.

NOTE — Syntans which are sensitive to boiling water should be dissolved at a lower temperature, and the temperature shall be stated in the report.

6.3 Liquid Syntans

Weigh out such a quantity of the material which will give as nearly as possible 1 litre of solution of required analytical strength (*see* **6.1**)· Add 400 ml boiling water and transfer to a 1 000-ml graduated flask. Add further boiling water to bring the volume to about 900 ml. Cool and make up the volume.

6.4 In the estimation of total solids, total solubles and nontannnis, the aliquots shall be maintained at *p*H 7·0 before evaporation over steam bath by the addition of required quantities of dilute standard sodium hydroxide solution. The necessary correction for the sodium hydroxide added should be applied to the mass of the dry residues while making calculations to determine the analytical characteristics.

7 DETERMINATION OF MOISTURE

7.1 Apparatus

7.1.1 *Air Oven*

Fitted with thermostatic control, capable of maintaining temperature at 100° ± 2°C.

7.1.2 *Wide-Mouthed Weighing Bottle*

7.2 Procedure

Transfer about 2 to 5 g of the finely ground material (*see* **5**) into a tared wide-mouthed weighing bottle and weigh accurately. Dry it at about 98·5° to 100°C in an oven for 3 to 4 hours, cool in a desiccator to room temperature and weigh again accurately. Repeat the process of drying and weighing until two weighings at an interval of one hour do not differ by more than 0·2 mg.

NOTES

1 Since for any syntan the sum of the moisture and the total solids is 100 percent, a determination of either is sufficient.

2 If the weight of the dried material is found to increase with longer drying, then the lowest mass found shall be taken.

7.3 Calculation

7.3.1 *Solid Syntan*

$$\text{Moisture, percent by mass} = \frac{(M_1 - M_2)}{M_1} \times 100$$

where

M_1 = mass in g of the material taken for the test, and

M_2 = mass in g of the residue left after drying

7.3.2 *Liquid Syntan*

$$\text{Moisture, percent by mass} = 100 - X$$

where

X = total residue, percent by mass.

8 DETERMINATION OF TOTAL SOLID

8.1 Apparatus

8.1.1 *Steam-Bath*

8.1.2 *Air-Oven*

Fitted with thermostatic control, capable of maintaining temperature at 100° ± 2°C.

8.2 Procedure

Evaporate to dryness on a steam bath 50 ml of the syntan solution prepared from a known amount of the material according to method prescribed in 6. Dry the residue at 98·5° to 100°C in an oven to constant mass. Consider the mass as constant when two weighings at an interval of one hour do not differ by more than 0·2 mg.

8.3 Calculation

$$\text{Total solid, percent by mass} = \frac{M_2}{M_1} \times 100$$

where

M_2 = mass in g of the material left after drying, and

M_1 = mass in g of the material taken for test.

9 DETERMINATION OF TOTAL SOLUBLES

9.1 Procedure

Filter the original solution (prepared as in **6**) through Whatmen filter paper (No. 1). Reject the first 150 ml of the filtrate and continue the filtration until it is clear from both transmitted and reflected light. Transfer 50 ml of the filtrate to a porcelain dish and evaporate to dryness. Dry the residue in an oven at 98·5 to 100°C for 3 hour and weigh to constant mass.

9.2 Calculation

$$\text{Total soluble, percent by mass} = \frac{M_2}{V_2} \times \frac{V_1}{M_1} \times 100$$

where

M_1 = mass in g of the material taken for the test,

M_2 = mass in g of the residue,

V_1 = volume in ml of the originally made up solution, and

V_2 = volume in ml of the solution taken for the test.

10 DETERMINATION OF NON-TANNINS

10.1 Principle

The non-tannins are determined from the matter left after the tannins are adsorbed by the hide powder.

10.2 Apparatus

10.2.1 *Glass Cylinder* — 500 ml capacity.

10.2.2 *Filtration Flask* — 500 ml capacity.

10.2.3 *Porcelain Dish* — between 7 to 8·5 cm in diameter, glazed inside.

10.2.4 *Steam-Bath*

10.2.5 *Vacuum Oven* — fitted with thermostatic control, capable of maintaining temperature at 100° ± 2°C.

10.3 Reagent

10.3.1 *Hydrochloric Acid* — (*see* IS 265 : 1987).

10.3.2 *Chrome Alum Solution*

Dissolve 30 g of the chrome alum in distilled water at room temperature and make up the solution to 1 000 ml. Chrome alum solution more than 30 days old shall not be used.

10.3.3 *Kaolin*

Take such quality of kaolin that when 1 g of it is suspended in 100 ml of distilled water and well shaken the *p*H of the suspension shall be between 4·0 and 6·0, that is, it shall neither give colour with methyl orange nor deep purple colour with bromocresol purple. When 1 g kaolin is shaken with 100 ml of 0·01 N acetic acid and the mixture filtered, the filtrate shall leave less than 1 mg of residue after evaporation and drying.

10.3.4 *Chromed Hide Powder*

For each analysis digest a multiple of the quantity of hide powder containing 6·25 g of dry matter, with ten times its weight of distilled water for one hour. Add to this 1 ml of chrome alum solution for each gram of air-dry hide powder taken, stir frequently for several hours and then allow to stand overnight. Next morning transfer the chromed powder to a clean linen, drain and squeeze. Place the cloth containing the powder in a suitable vessel (an enamel bucket is suitable for large quantities), open out the cloth bag and pour on the powder a quantity of water equal to 15 times the weight of the air-dry hide powder taken. Mix the powder and water thoroughly and digest for 15 minutes, after which lift out the cloth and powder and immediately drain and squeeze the powder so that it contains approximately 75 percent moisture. Digest the powder three times more in the same way with distilled water. Thoroughly break up the cake of chromed powder, mix until uniformly free from lumps and weigh the whole mass.

10.3.5 *Linen*

Pieces of linen cloth shall be used for washing the chromed hide powder and for the preliminary filtration to detannized solutions. The linen shall be freed from weighting matter by boiling in several changes of distilled water.

10.3.6 *Hide Powder*

It shall comply with the following requirements:

a) The ash content of the powder shall be not more than 0·3 percent by mass.

b) When 7 g of the air-dry powder is allowed to stand in contact with 100 ml of 0·1 N potassium chloride solution, previously adjusted to *p*H 5·5 with 0·01 N acetic acid for 24 hours, then the liquor obtained by centrifuging or by filtering through a paper previously washed with 0·1 N potassium chloride solution, shall have a *p*H ranging between 5·0 and 5·5.

10.3.7 *Gelatin-Salt Reagent*

Dissolve 1 g of photographic grade gelatin and 10 g of pure sodium chloride in 100 ml of distilled water at a temperature not higher than 60°C and adjust the *p*H to 4·7 approximately by adding acid or alkali, that is, the solution shall give red colour with methyl red and yellow with methyl orange. To preserve this solution, add 2 ml toluene (IS 1839 : 1961) or preferably, a few crystals of *p*-dichlorobenzene and keep in a cool place. Freshly prepared solution is preferable.

10.4 Procedure

Weigh accurately a quantity of wet-hide powder (say *X* g) containing about 6·25 g dry powder (*see* **10.3.4**) and add it immediately to 100 ml of the unfiltered syntan solution (prepared as in **6**) plus (26·25 − *X*) ml of distilled water, already present in a wide mouthed bottle of 150 to 300-ml capacity. Stopper the wide mouthed bottle tightly with a rubber bung, and shake it vigorously first by hand for 15 seconds and then transfer to a mechanical rotary shaker and shake for exactly 10 minutes at 50 to 65 rpm. Pour the powder and the solution on a clean, dry linen filter cloth supported by a funnel, drain and squeeze by hand. Add to the filtrate 1 g of kaolin and pour into a single 15 cm pleated filter paper returning the filtrate repeatedly until it is clear. Keep the funnel and collecting vessel covered during filtration. Test the filtrate with gelatin-salt reagent and if 10 ml gives any turbidity with 1 or 2 drops of the reagent, mention this in the report of the analysis. Evaporate in a tared porcelain dish 50 ml of the filtrate and dry the residue in a vacuum oven at 98·5° to 100°C, cool and weigh until constant mass is obtained.

NOTE — To correct for the 20 ml of water of dilution introduced by the wet hide powder into the 100 ml of tannin solution, the residue mass is multiplied by 1·2. This corrected mass is the residue from 50 ml of original analytical solution.

10.5 Calculation

$$\text{Non-tannins, percent by mass} = \frac{M_2 \times V_1}{M_1 \times V_2} \times 100$$

where

M_2 = mass in g of the residue left after drying,

V_1 = volume in ml made up originally,

M_1 = mass in g of the material taken for the test, and

V_2 = Volume in ml of the test solution taken.

11 DETERMINATION OF TANNIN

11.1 Principle

The tannins are determined by finding the difference between the percentages of total solubles and the non-tannins.

11.2 Procedure

11.2.1 Determine the total solubles of the syntan according to the method given in **9**.

11.2.2 Determine the non-tannins present in the syntan according to the method given in **10**.

11.3 Calculation

Tannins, percent by mass = $X - Y$

where

X = total soluble, percent by mass (**11.2.1**), and

Y = non-tannins, percent by mass (**11.2.2**).

NOTES

1 All analyses shall be done in duplicate. The aboslute error in tannin content in duplicate analyses should not be more than 2 percent.

2 Where analyses are carried out by different chemists on the same sample, the result shall not differ by more than 3 percent of the total tannin content.

12 DETERMINATION OF INSOLUBLES

12.1 Principle

Insolubles are determined from the difference between the percentage of total solids and total solubles.

12.2 Procedure

12.2.1 Determine the total solid according to the method prescribed in **8**.

12.2.2 Determine the total solubles according to the method prescribed in **9**.

12.2.3 Determine moisture in accordance with the method prescribed in **7**.

12.3 Calculation

Insolubles, percent by mass = $X - Y$

where

X = total solids, percent by mass (**12.2.1**); and

Y = total solubles, percent by mass (**12.2.2**).

13 DETERMINATION OF *p*H

13.1 Apparatus

13.1.1 p*H Meter* — with glass electrode.

13.2 Procedure

Determine the *p*H of the solution (prepared as in **6**) adjusting the relative density to 1·05 at 27°C with cold water, by using a suitable *p*H meter.

14 DETERMINATION OF COLOUR

14.1 Apparatus

14.1.1 *Lovibond Tintometer* — with artificial light source.

14.2 Procedure

Fill the solution (prepared as in **6**) immediately after filtration in a 1-cm cell and determine the colour value. Calculate the result on 0·5 percent tannin content basis and express in units or red and yellow.

15 DETERMINATION OF IRON

15.1 Reagents

15.1.1 *Fuming Nitric Acid*

15.1.2 *Bromine Water* — saturated.

15.1.3 *Concentrated Hydrochloric Acid* — (*see* IS 265 : 1987).

15.1.4 *Ammonium Hydroxide* — 1:1 (*v/v*).

15.1.5 *Dilute Hydrochloric Acid* — 1:1 (*v/v*).

15.1.6 *Dilute Sulphuric Acid* — 1:20 (*v/v*).

15.1.7 *Potassium Permanganate Solution* — approximately 0·1 N.

15.1.8 *Potassium Thiocyanate Solution*

Dissolve 10 g of potassium thiocyanate (or ammonium thiocyanate) in 100 ml of water.

15.1.9 *Stock Iron Solution*

Dissolve exactly 0·702 g of pure ferrous ammonium sulphate [$FeSo_4$ (NH_4)$_2$ SO_4, $6H_2O$] in 50 ml of water and 20 ml of dilute sulphuric acid (1:4) and add 0·1 N potassium permanganate solution drop by drop until a faint pink colour is obtained. Adjust the volume to one litre with water. The strength of this solution is ten times that of the standard solution. The keeping quality of this solution is better at this strength. Store this solution and use it when required.

15.1.10 *Standard Iron Solution*

Prepare a fresh solution by diluting 10 ml of the stock solution (**15.1.9**) to 100 ml with water. One millilitre of this solution contains 0·000 01 g of iron (as Fe).

15.2 Procedure

15.2.1 Weigh accurately a quantity of material which is equivalent to 5 g on moisture-free basis, to the nearest 0·1 mg in a glazed flat-bottomed porcelain basin. Heat it first over a low flame until the residue is completely charred and ceases to give out smoke. Then place the basin in an electric muffle furnace (600°C) to make it free of carbon.

15.2.2 In case all the carbon is not removed, take out the basin from the furnace and allow it to cool. Add a little hot water and break up the carbonaceous matter with a glass rod. Extract it with water by warming it over a water-bath for a few minutes. Decant the supernatant liquid, filter it through an ashless filter paper and collect the filtrate. Repeat the process of extraction and filtration thrice. Combine the filtrates. Transfer the residue on the filter paper and wash it several times with hot water and add the washings to the fitlrate. Place the filter paper and the contents in the original basin, dry, ignite and ash as before, until all the carbon is burnt off. Cool the basin again and transfer the combined filtrates and washings quantitatively to it. Evaporate the extract to dryness on a water-bath. Replace the basin in an electric muffle furnace (600°C).

15.2.3 Cool the basin and moisten the carbon-free ash with hot water. To this add five drops each of fuming nitric acid and bromine water. Heat slowly until the fumes cease to come out. Evaporate to dryness. Moisten the residue with water and digest it with 5 ml of concentrated hydrochloric acid over a water-bath for a few minutes, dilute with water and filter into a 25-ml beaker. Adjust the volume to approximately 75 ml (by boiling, if necessary) and make it faintly alkaline with ammonium hydroxide. Boil off the excess ammonia gently and allow the precipitated iron and aluminium hydroxides to coagulate.

15.2.4 Allow the solution to stand for few minutes until the precipitate settles. Decant the supernatant liquid quickly through an asbestos Gooch filter previously washed with hot dilute hydrochloric acid, water, ammonium hydroxide and water successively. Wash the precipitate three times with hot water, made faintly alkaline with five drops of ammonium hydroxide per litre. Transfer the precipitate quantitatively to the filter and wash it four times with hot faintly ammonical water. Keep the combined filtrate and washings for the determination of copper (*see* **16**).

15.2.5 Transfer 10 ml of dilute sulphuric acid and 5 ml or potassium thiocyanate solution to each of the two identical 50-ml Nessler cylinders. To one of the Nessler cylinders add 10 ml of the made up solution (*see* **15.2.5**) and make up the volume to mark. From a burette, run in standard iron solution to the second Nessler cylinder until the tints of the two cylinders are identical when viewed vertically through the depths of the cylinders, the cylinders being held over a white surface.

15.3 Calculation

Calculate the iron content of the sample from the known volume of the standard iron solution required for matching.

16 DETERMINATION OF ASH

16.1 Procedure

Heated gently about 2 g of the syntan in a silica crucible on a Bunsen Burner till ashed. Transfer the crucible to a muffle furnace and maintain the temperature at 800°C for three hours. Repeat the procedure till constant mass is obtained.

16.2 Calculation

$$\text{Ash, percent by mass} = \frac{M_1 \times 100}{M}$$

where

M = mass in g of the sample taken for test, and

M_1 = mass in g of the residue obtained.

17 REPORTING THE RESULTS

17.1 The results shall be reported to one place of decimals only.

17.2 All analyses shall be made in strict accordance with the methods prescribed and the report shall state:

a) that the analyses has been according to the methods prescribed in Indian Standard;

b) whether the tannin infusion has been filtered or not;

c) whether kaolin has been used for the filtration of the syntan solutions;

d) the batch number of hide powder;

e) whether moisture has been determined by direct drying; and

f) if water at a temperature lower than boiling point is used for the preparation of the analytical solution (*See* **6.2**), the temperature shall be stated.

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